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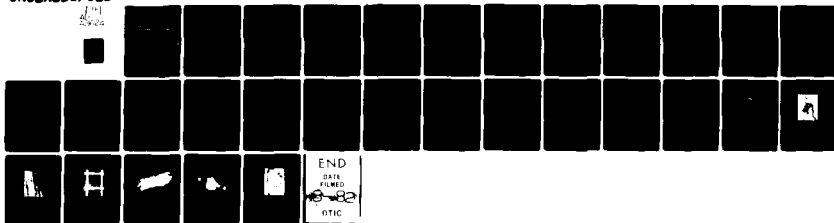
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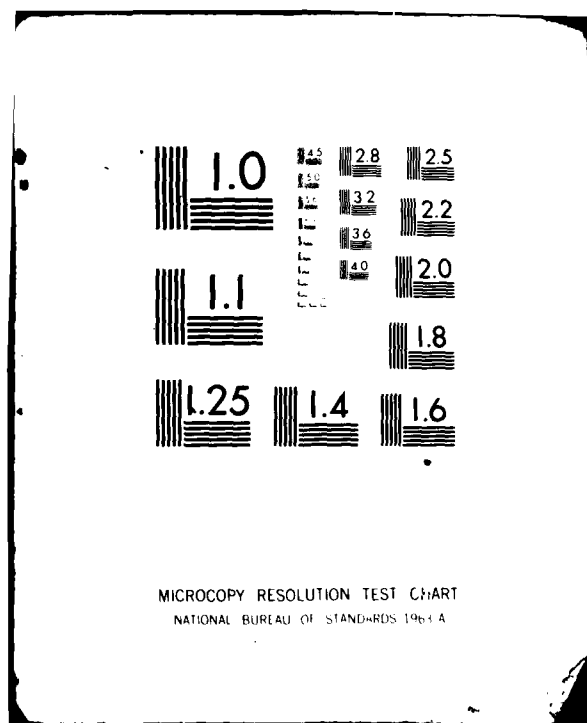
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INTERDISCIPLINARY RESEARCH PROJECT ON SLIDING WEAR

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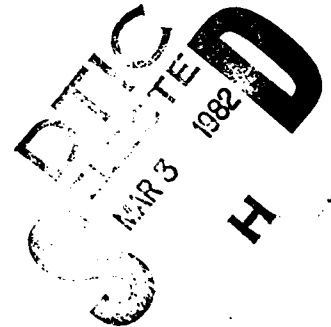
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RF Projects 761143/711174
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Final Report



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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) This report summarizes basic research on the mechanisms of sliding friction and wear done jointly by groups at The Ohio State University and Battelle Columbus Laboratories. The OSU portion of this interdisciplinary work has been concerned with unlubricated sliding wear. At Battelle the work has included sliding tests with selected lubricants, <u>in situ</u> sliding in an SEM, and application of fracture mechanics to sliding wear.		

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I. INTRODUCTION

This project was concerned with the basic mechanisms of sliding friction and wear. The personnel included researchers familiar with friction, lubrication, wear, mechanics, fracture, materials science, and various analytical techniques. The Ohio State portion of this interdisciplinary research concentrated on unlubricated sliding with an emphasis on materials aspects. Materials were chosen to test the validity of available models for the generation of wear debris, especially the commonly observed wear debris flakes. At Battelle the work included sliding tests with selected lubricants, in situ sliding in an SEM, and application of fracture mechanics. Similar materials, test equipment, and experimental conditions were used for the Ohio State and Battelle research to facilitate comparison. The test samples and the debris were analysed by using a wide range of techniques ranging from optical metallography and microhardness testing to SEM/EDAX and TEM analysis. Changes in the surfaces and in the sub-surface materials were studied.

The most widely accepted explanation for friction and wear behavior emphasizes adhesive interactions of surface asperities. This approach has led to an emphasis on surface chemistry and on continuum mechanics, with much less attention paid to the structures of the interacting materials themselves. The delamination ideas of Suh helped to stimulate renewed interest in the mechanisms of sliding friction and wear. Our own approach has been to emphasize plastic deformation and fracture in near-surface material. Therefore we expected that materials properties

which influence plastic deformation and fracture would be important for friction and wear.

When our work on these projects began, we decided that it would be useful to compare the results of unlubricated and lubricated sliding tests using similar materials, equipment, and conditions. We also wanted to obtain data which could explain the familiar observation that sliding wear debris particles are commonly flakes. Such data could provide evidence for or against Suh's reports that sub-surface cracks in the sample material nucleate and grow to give flake debris by a delamination process.

In the following sections we summarize our observations. The results do not support a simple delamination model for wear during either unlubricated or lubricated sliding. We do find evidence that wear mechanisms are similar for both kinds of tests. However, we also report evidence that more than one mechanism can operate in both cases.

II. OHIO STATE WORK

1. Experimental Procedures: Most of the unlubricated sliding tests were done with an LFW block-on-ring machine made by Faville-LeValley Corp. We modified this machine by attaching a plexiglass box to the front to allow control of test atmosphere.

A typical test used the following conditions. The atmosphere was dry argon with a relative humidity reading of 20%. Sliding speed was kept low (5 cm/sec) to assure that average surface temperature was not excessive. Near-surface temperature and friction force were monitored on a chart recorder. The test rings were usually 440C martensitic stainless steel (for the Cu-Ni series, Cr-plated rings were used for some of the tests). Loads were in the range 15-45 lb. (6.8 kg-20.4 kg), and sliding times were typically two hours. Debris particles were collected at various times during sliding. Details of specimen preparation before testing and specimen and debris analysis after testing are described in the enclosed publications.

2. Cu-Ni Alloys: Our studies of a series of Cu-Ni alloys (M.S. Thesis, J. Schell, OSU, 1979) were particularly important for identifying important phenomena affecting friction and wear. In the enclosed paper on these materials one can follow the development of our thinking as we acquired data. Our conclusions and our initial expectations were quite different.

We selected the Cu-Ni system because we could work with a single FCC phase for which hardness and stacking fault energy (SFE) data were

available. Since these properties vary strongly but differently with composition, we thought that our results could be used to determine which of these properties might be more important for sliding behavior.

Our first results seemed to show that formation of NiO increased the wear rate at higher Ni concentrations by embrittling the surface material. This effect may contribute, but we later found that another effect was more important. A layer of ultra-fine structured transfer material gradually accumulates on the surface. The degree of transfer depends on the sample composition. The transfer patches or layers include material from both the ring and the block, intimately mixed in a micro-composite. When the Ni content and the amount of transfer material exceed critical levels, significant ring damage occurs and the system behaves erratically. Analysis with an EDAX-equipped SEM was particularly useful for characterizing the Cu-Ni specimen and providing evidence for the importance of transfer material during sliding.

3. Austenitic Stainless Steels: Results obtained during an earlier project on these materials (M.S. Thesis, K. L. Hsu, OSU, 1978) showed that the relative stability of the austenite to the formation of strain-induced martensite significantly affected sliding behavior. For example, α' martensite formed readily in the less stable 304 alloys and this led to formation of irregular chunky debris by fracture in the hard surface layer. In contrast, less α' formed in samples of 316, and the debris were larger and consisted mostly of austenite.

V. Shende extended this work (M.S. Thesis, OSU, 1980) for two reasons. One was to test additional materials chosen on the basis of

their relative austenite stabilities rather than their SFE's. The other reason was to obtain data which could allow us to distinguish between strain-induced martensite and material transferred from the BCC test rings. Since the lattice parameters and compositions of the α' and the rings were similar, we also performed some self-mated tests to be sure that the modified surface material was indeed strain-induced martensite.

The alloys tested were 304, 305, 316, 310, and 332. It was concluded that both strain-induced martensite and transfer material formed during sliding of these materials against 440C. The most wear-resistant alloy was 310, and it was also the most stable alloy tested.

4. Cu-Si and Cu-Ge: This work (M.S. Thesis, N. Haddad-Kaveh, OSU, 1981) was designed to complement other research on Cu-X solid solutions.* For similar atomic fractions of solutes (or, e/a ratios), we expected that the similar stacking fault energies would influence deformation and fracture in similar ways by affecting such processes as cell formation and twinning. However, we also expected differences in sliding behavior related to the different oxidation resistance of these alloys. Materials tested were Cu with 1, 3, and 5 at. % Si, 1, 3, and 5 at. % Ge, and 8.8 at. % Ge. Some were produced by Olin Corp. for our work, and some were produced in our laboratory. Different grain sizes and hardnesses were

*For another project we tested Cu, Cu-Zn, Cu-Al, and Cu-Al alloys (unlubricated). The Cu-X alloys used for the lubricated tests at Battelle were the same materials.

obtained by thermomechanical treatments. Test rings were 440C stainless steel.

The resulting friction and wear data showed wide variations which did not correlate in any simple way with either the type of solute or the composition. The amount of transfer material on the sample blocks increased as the % Si increased, but it decreased as the % Ge increased. There was more transfer at the entrance side of the wear scar than at the exit side. Annealed samples provided smaller fluctuations in the friction trace, but finer grain sizes also provided sharper traces.

As we have found with several other materials systems, the transfer material was composed of small pieces of block material and ring material intimately mixed in a micro-composite (with possibly a small amount of oxide as well). The color changes observed during sliding of copper alloys against ferrous counterfaces may also be related to the appearance of transfer material on the surface rather than caused by composition changes in the near-surface sample material itself.

5. Transfer: We are now quite certain that transfer layers are an important aspect of the sliding wear of metals. To understand sliding wear, we must first understand how these layers and their small (100-300 Å) particles are formed. At this stage, one can imagine a number of possible sequences involving deformation, adhesion, fracture, oxidation, etc., but existing data are not sufficient for choosing one set of mechanisms over another.

The work on the Cu-Ni alloys has indicated that adhesion is probably involved in the formation of transfer layers. It seems very

likely that adhesion is also involved in the formation of the very small particles which compose the transfer layer. Thus, we acknowledge an important role for adhesion in sliding wear mechanisms, but we believe that available data show that the role of adhesion is more complex than that described in traditional theories of "adhesive wear".

We have made preliminary calculations to predict the tendency of one metal to adhere to another and form a transfer layer. An important assumption is that adhesion will lead to fracture at weak locations such as dislocation walls generated by extensive plastic deformation. The approach used involves comparison of adhesion energies and boundary or wall energies for the two interacting materials. The energies can be roughly estimated by any of several related models, including a simple pair-bonding model (L. H. Chen, M.S./PhD candidate).

The models also allow consideration of changes in transfer due to changes in composition. Thus, we can predict whether a given solute will increase or decrease the tendency of a metal at an asperity to fracture and adhere to the opposing surface. We have used the results of our calculations to select an improved ring material for LFW block-on-ring sliding tests. We had used 440 C steel rings for tests of copper alloy sample blocks, but the results were complicated by significant amounts of damage to the ring related to transfer of iron to the copper alloy. Since we predicted that Mo would reduce this effect, test rings containing molybdenum were used. The result was definite reduction in ring damage, although some transfer still occurred.

Research on this important topic will continue under the sponsorship of ONR.

III. BATTELLE WORK

A. LUBRICATED WEAR STUDIES

1. Wear Tests: Short term wear experiments have been conducted using the LFW-1 ring and block machine and selected liquid lubricants. The effects of boundary lubrication on the wear process and debris generation have been investigated.

The following operating conditions were used in the experimental work:*

Load	30 lbs (13.6 kg)
Speed	35 RPM
Ring Diameter	1.4 in. (3.6 cm)
Surface Velocity	12.8 fpm (6.5 cm/sec)
Contact Geometry	Flat on ring (line contact)
Environment	Dry argon gas (30 - 40 % RH)
Length of run	25 - 30 minutes
Lubricants	1% solution of stearic acid in Octadecane; Dibasic acid ester (Mil-1-7808 oil); Dow Corning DC 200 (Dimethyl silicone)
Wear block materials	Cu + 3.2 wt. % Al Cu + 7.5 wt. % Al Cu + 15 wt. % Zn

Lubricants of low viscosity were selected and operating conditions of slow sliding speed with line contact were selected in order to minimize hydrodynamic effects in the contact area.

* A full description of the apparatus and test method will appear in a forthcoming paper to be given at the 1982 Joint ASME-ASLE Conference in the Fall of 1982 in Cincinnati.

After installation of the ring and block specimens, the apparatus enclosure was sealed and dry argon gas flow initiated. When the relative humidity reached 30 - 40%, lubricant was applied to the ring with a syringe which penetrated the sealed enclosure. Sufficient lubricant was applied so that a permanent drop clung to the bottom of the ring. With the dead weight load applied the ring was started rotating at speed. From the initiation of rotation, the lubricant drop was observed with low power magnification and the appearance of the first metallic wear debris looked for. The induction period was timed and the morphologies of the first pieces of wear debris were noted. Changes in the character of the debris with increasing time elapse were also noted. Upon completion of the 30 minute run, the lubricant drop was removed and the debris it held was extracted for analysis.

The wear scars on the block and on the ring surface were examined by light and scanning electron microscopy. The debris from the lubricant were examined and analyzed in the Scanning Transmission Electron Microscope (STEM).

2. Experimental Results: Wear Scars: The wear scar microtopography was similar for the three copper base alloys investigated. Some differences were noted in the effect of lubricant species. The basic morphology of the wear microtopography consisted of long striations, parallel to the sliding direction and having a distribution of sizes. The average width of a striation was 15 μm . The depths averaged about 0.5 μm . A typical wear surface is shown in figure 1. Note that in addition to striations, there are smeared over areas later identified

as debris agglomerations. At the inlet and exit borders of the wear scar, metal extrusion was always found. This is shown in figure 2. Transfer to the ring from the block always occurred. All of these features were similar to those observed at OSU during work on unlubricated sliding.

Compared with the boundary lubricated tests, the extent of smeared material in the wear surfaces increased when the silicone lubricant was used. Much of the metal flow occurred as striation ridges were flattened out. An example of a typical wear scar observed for silicone lubrication is shown in Figure 3. Transfer to the steel ring was heavier when the silicone was used. Transfer lumps on the ring appeared to increase the severity of wear marks on the block.

Debris: Wear debris extracted from the lubricant were composed of a mixture of relatively large bright flakes which appeared early in the run and very small rounded particles. The flakes ranged in size from 10 to 30 μm while the small particles ranged from 200 \AA to 1000 \AA in size. The very small particles were always associated with a gel-like substance presumed to be organic. This material did not show any recognizable pattern in electron diffraction analysis. The small debris, recognized by an increase in cloudiness of the lubricant drop, always appeared some time after the appearance of the large shiny particles. Infra-red analysis of the gel developed during the experiments using stearic acid lubricant showed strong indication of metal soap and very weak indication of stearic acid (the acid had reacted for the most part).

Examples of the large particles are shown in figure 4. The particles are distributed on a grid photographed in the light microscope. The color of the particles was yellow, i.e., similar to the alloy color. The small particles were embedded in colonies dispersed in a piece of gel. Examples of this structure are shown in figures 5 and 6. Stereo photomicrographs revealed the structure to consist of gel-coated particles agglomerated into colonies. Particle analysis by electron diffraction and EDAX indicated the presence of the elements of the wear block alloy and faint indications of iron (from the ring).

In Situ SEM: In a related investigation, a pin on disk device was constructed and inserted into an SEM for observation of the disk surface during sliding. Evidence of plastic deformation could be seen during the first pass. In subsequent passes, the evolution of surface micro-topography could be observed. For example, lateral flow of $\sim 12 \mu\text{m/pass}$ was measured locally on a copper disk with a 52100 steel pin.

3. Discussion of the Results: Microscopic details of the wear scars suggest that heavy deformation is occurring to a depth of under a micron and that the deformation and striations are caused by asperities in the ring surface. These are from the grinding pattern (from machine finish) and from transfer lumps from the block. Some wear debris are generated by lateral extrusion of striations (this was also seen in in-microscope SEM wear experiments)*. The large flakes have been

*The experiments have been described in a paper given at the ASM Materials Science Seminar and in a paper to appear in a forthcoming issue of WEAR.

identified as coming from the extrusions at the entrance and exit boundaries of the wear scar. For the test geometry used in these experiments the debris was carried into the entrance wedge of the contact area and was compressed into larger particles which adhered to the block surface. The extrusion and break off of extruded material occurs early in the wear process as implied by the initial appearance in the drop of lubricant. A piece of broken off extrusion is shown in figure 7. The induction period always observed prior to detection of the first particles implies that time is required to build up the extrusions and break them off.

The origin of the very small particles is not obvious. The possibility that the small particles come from chemical reactions between lubricant constituents (selective attack along cell walls) seemed to be disproved by the similar results of the silicone oil experiments and the boundary lubricant experiments.

It was not surprising to find a metal soap associated with wear debris. Certainly, the size and shape of the particles (rounded) suggest chemical reaction. The reaction could occur after the formation of the particle with the result that each particle would be encased in a jacket of reaction product. The fact that gel-particle colonies contained metal particles dispersed throughout and not concentrated on one surface indicated that the reaction took place after the formation of the particle. If corrosive removal of metal were occurring, one might expect a surface film of soap to grow on the block and then be stripped off with metal particles embedded in the interface zone.

Further, the fact that an inert fluid like DC200 silicone produced a gel was surprising and made the reaction thesis more difficult. Gelation in silicones can be caused by oxidation of the fluid at elevated temperatures. Dimethyl silicone will decompose at 315°C and will begin to form a gel at 250°C. With small metal particles present, a gel might form at lower temperatures. It is possible that local surface temperatures could reach gel formation conditions - enough to produce small quantities of the gel. Since the very small debris particles always appeared much later than the large particles, the time for sufficient oxidation of the lubricant may be reflected in this phenomenon. If the gel tends to nucleate around minute metal particles, the particles must have been present prior to oxidation (they may not become visible in the lubricant until they become coated and form colonies). It is also possible that oxide formed at frictionally heated contacts may fracture and be released as tiny particles.

4. Discussion: The effect of lubricants on the wear process of ductile copper-base alloys is to influence the extent of metal transfer and the dispersion of wear debris. Those lubricants with known boundary lubricating properties reduce the severity of metal transfer to the harder surface (steel). The topographical changes in the steel surface are not as marked as found in unlubricated sliding and therefore the amount of surface deformation in the ductile material is moderated. The alloy content of the copper base alloys appeared to have little influence on the microtopography developed during lubricated wear.

A poor boundary lubricant such as a dimethyl silicone increases the extent of transfer and the depth of damage in the surface. This suggests that metal adhesion during the transfer process is reduced by the use of a boundary lubricant.

Even with a boundary lubricant, however, debris is generated. With ductile materials like the alloys studied, metal smearing and extrusion occur in spite of lubrication. The particle-gel component of the debris is present with all lubricants used and the particle-gel complexes look similar.

The fluid phase of the lubricant influences the distribution of the wear debris as it is released from the surface. Debris is dispersed in the liquid phase and the very small particles tend to follow the flow lines as the lubricant is drawn into the contact region. The way in which recycled debris is redeposited and compacted onto the wear surfaces will be influenced by the flow and dispersal process. Turbulance at the entrance region will tend to separate smaller mass particles and keep them out of the interface. The higher the surface velocity, the larger the particles that will be influenced in this way.

The existence of extremely small debris particles (in the hundreds of Angstroms) was not anticipated in the beginning of the study. How they are generated is not yet determined. However, since the same scale constituents have been found in the unlubricated wear studies, the processes involved in their origin are important for understanding the mechanisms of both lubricated and unlubricated wear.

B. THE FRACTURE MECHANICS ASPECTS OF WEAR

At the outset of the research modelling of sliding wear was limited to linear elasticity. The experimental evidence, in contrast, convincingly demonstrated that the phenomenon is overwhelmingly plastic. For this reason the research was aimed toward development of a plastic analysis.

The initial stage was formulation of a base-line elastic model. It consisted of a subsurface crack under the influence of a line force representing an asperity. The major conclusion was that the most significant driving force for the wear process is the combined shear/compression stress field ahead of the asperity. Further development of the elastic model showed that a crack parallel to the wear surface is not stable but will turn toward the surface.

Incorporation of plasticity was done via a strip-yield model. Among other things this model accounts for step changes in wear rate with increasing load.

The final model was based on observations of subsurface morphology of wear specimens which had experienced sliding. These observations suggest that wear takes place in a small layer at the surface which was denoted the process layer in analogy with the crack tip process zone in fracture mechanics. This model also provides a relation for sliding wear rate analogous to Archard's law for wear:

$$W = k_r F / \sigma_f ,$$

where w is the wear rate, F is the normal force and σ_f is the flow stress within the process zone.

IV. SUMMARY

1. Both unlubricated and lubricated sliding wear involve plastic deformation and fracture processes.
2. The wear mechanisms appear to be similar for lubricated and unlubricated sliding.
3. The plastic strains are very large near the sliding interface for both lubricated and unlubricated sliding.
4. The development of transfer material and layers affects both friction and wear for lubricated and unlubricated sliding.
5. Debris particles have fine substructure, often in the size range 100 - 300 Å.
6. For copper and copper alloys on steel both the transfer material and the debris have mixed composition (i.e., both Cu and Fe).
7. We did not see evidence of simple delamination processes in which sub-surface cracks nucleated and grew to give loose debris particles. Some debris flakes seem to arise from fractured extrusions (at ends of scar and laterally at ridges) while others seem to be associated with delamination of transfer material. No single mechanism is adequate to explain observations.

V. Participating Scientific Personnel

1. David A. Rigney, Professor, Metallurgical Engineering, The Ohio State University.
2. William A. Glaeser, Tribology Section, Battelle Columbus Laboratories.
3. A. R. Rosenfield, Metal Science, Battelle Columbus Laboratories.
4. Glyn Meyrick, Professor, Metallurgical Engineering, The Ohio State University (temporary during part of 1981, while D. Rigney was on leave at Cambridge University).
5. Jerry D. Schell, M.S. Degree, Met. E., OSU, 1979. Thesis Title: Microstructural Aspects of the Unlubricated Friction and Wear of Cu-Ni Alloys.
6. Vijay A. Shende, M.S. Degree, Met. E., OSU, 1980. Thesis Title: A Study of the Effect of Metastability on the Unlubricated Sliding Wear of Austenitic Stainless Steels.
7. Nasrollah Haddad-Kaveh, M.S. Degree, Met. E., OSU, 1981. Thesis Topic: Sliding Friction and Wear Behavior of Cu-Si and Cu-Ge Single Phase Alloys.
8. Li-Hui Chen, M.S. candidate (possible PhD candidate), Met. E., OSU. Topic: Metal Transfer during Sliding (theory and experiments).

VI. LIST OF PUBLICATIONS DURING PROJECTS

1. D.A. Rigney, International Conference on Fundamentals of Tribology, MIT, June, 1978, Proceedings, Discussion Paper on "Mechanical Properties of Near-Surface Material in Friction and Wear," by A.S. Argon; book, Fundamentals of Tribology, eds., N. Suh and N.Saka, MIT Press, 1980.
2. D.A. Rigney and J.P. Hirth, Int'l. Conf. on Fundamentals of Tribology, MIT, June, 1978, Proceedings, "Plastic Deformation and Sliding Friction of Metals," (short version); book, Fundamentals of Tribology, eds. N. Suh and N. Saka, MIT press, 1980.
3. D.A. Rigney and J.P. Hirth Wear, 53, 345-370, 1979, "Plastic Deformation and Sliding Friction of Metals." (long version)
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5. D.A. Rigney, "Dislocation Content at Large Plastic Strains," Scripta Met. 13, 353-354, (1979).
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7. J. Schell, P. Heilmann, and D.A. Rigney, "Friction and Wear of Cu-Ni Alloys," Int'l. Conference on Wear of Materials, III, San Francisco, March 30-April 2, 1981. (also Wear 75, [2], 205-220 [1982]).
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11. A.W. Ruff, L.K. Ives, and W.A. Glaeser, Characterization of Wear Surfaces and Wear Debris, 1980 ASM Materials Science Seminar, Pittsburgh; in Fundamentals of Friction and Wear of Materials, ed., D.A. Rigney, ASM, 1981.

12. W.A. Glaeser, Wear Experiments in the Scanning Electron Microscope, Wear (1982).
13. A.R. Rosenfield: "A fracture Mechanics Approach to Wear" Wear, 61 (1980) 125-132.
14. A.R. Rosenfield: "Wear and Fracture Mechanics" in D.A. Rigney, ed Fundamentals of Friction and Wear of Materials ASM .
15. A.R. Rosenfield: "A dislocation Theory Approach to Wear" Wear, 72 (1981) 97-103.
16. A.R. Rosenfield: "Elastic-Plastic Fracture Mechanics and Wear" Wear, 72 (1981) 244-253.

* * *

Also, we took part in two workshops organized by our sponsors (All Navy Tribology Workshop, Annapolis, May 22, 1979, and Materials Aspects of Wear, ARO/ONR at NBS, October 2, 1979. In addition, we presented many talks on this research.

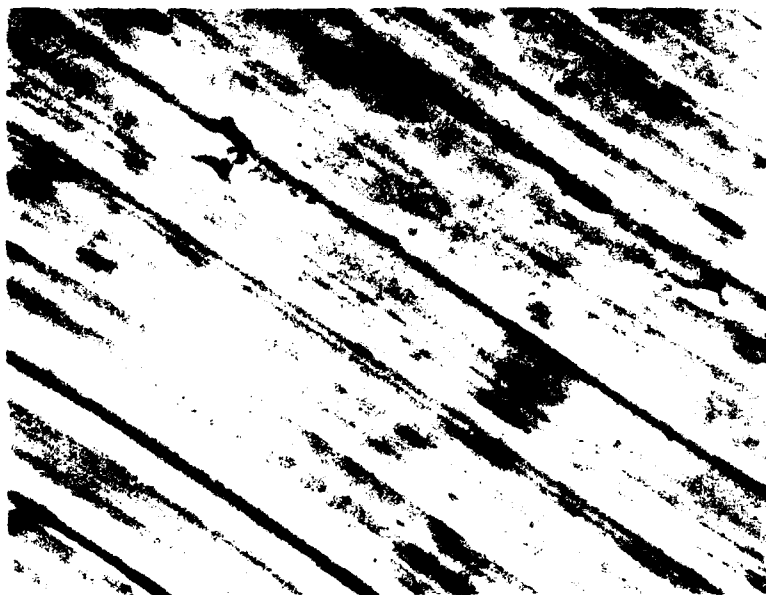


Figure 1. SEM micrograph of wear scar on a 15 Zn-Cu wear block,
lubricated with a 1% solution of stearic acid in octadecane.
(1600x)



Figure 2. Extrusion of surface material at the boundary of a wear scar on a 7.5 Al-Cu alloy block lubricated with a 1% stearic acid solution in octadecane. (570x)



Figure 3. SEM micrograph of wear scar on 3.2 Al-Cu alloy lubricated with DC 200 silicone oil. (2000x)



Figure 4. Light micrograph of wear debris deposited on a grid for STEM analysis. Debris is from a lubricated wear experiment using 1% stearic acid in octadecane on a 3.2 Al-Cu block. (500x)



Figure 5. TEM micrograph of a piece of wear debris from a lubricated wear experiment using 1% stearic acid in octadecane on OFHC copper block.



Figure 6. TEM micrograph of a piece of wear debris from a lubricated wear experiment using 1% stearic acid in octadecane on 3.2 Al-Cu alloy.



7. SEM micrograph of large wear fragment presumed to be a broken out extrusion (7.5 Al-Cu alloy lubricated with 1% stearic acid in octadecane). (540x)

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